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NOTE ON THE PREPARATION OF MANNOSE

BY

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Bureau of Standards

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NOTE ON THE PREPARATION OF MANNOSE

By E. P. Clark

ABSTRACT

A method is described by which mannose may be prepared easily and economically. Ivory-nut shavings or sawdust are treated with dilute NaOH, washed, and dried. Five hundred grams of the material thus prepared are digested for a day with 75 per cent sulphuric acid, then dissolved in water to make 5.5 liters. This mixture is boiled for two and one-half hours, neutralized with $BaCO_3$, concentrated, and the sugar crystallized from glacial acetic acid, giving a yield of 42 to 45 per cent of the treated meal.

A method for the preparation of mannose from ivory-nut shavings has recently been described by Horton.[1] In his procedure the shavings are first treated with dilute sodium-hydroxide solution, whereby gums and extractives are removed. This preliminary step is a great improvement over previous methods, as it gives a product of higher purity and eliminates the disadvantage of excessive foaming when the solution is concentrated. However, the mass of detail prescribed for subsequent steps offsets the advantage gained; hence the method, as a whole, is no improvement over that of Hudson and Sawyer.[2]

The writer has on various occasions prepared mannose by a very simple process which, when applied to ivory-nut shavings that have first been treated with sodium hydroxide, gives a yield that is considerably higher than either author has reported. This method is given below, as its simplicity and economy will appeal to workers who have to prepare this sugar.

Sifted ivory-nut shavings are added to 10 times their weight of boiling 1 per cent sodium-hydroxide solution. The mixture is at once removed from the source of heat and stirred occasionally during one-half hour. The shavings are then washed thoroughly with running water until neutral and clear, and dried.

Five hundred grams of the material thus prepared are thoroughly mixed with 500 g of 75 per cent sulphuric acid and allowed to stand until the next day. This mass is dissolved in water, making a volume of 5.5 liters, and boiled under a reflux for two

[1] Horton, Paul M., J. Ind. and Eng. Chem., 13, No. 11, p. 1040; 1921.
[2] Hudson C. S., and Sawyer, H. L., J. Am. Chem. Soc., 39, p. 470; 1917.

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and one-half hours. While the liquid is still boiling, it is neutralized with a thin paste of precipitated barium carbonate. The solution is at once filtered through a thin layer of active carbon placed on moistened filter paper in a Büchner funnel. The filtrate generally contains a little barium, probably in combination with organic acids. This is removed by adding a few cubic centimeters of dilute sulphuric acid until no further precipitate is formed. The barium sulphate is filtered off and the solution evaporated under reduced pressure to 87 to 88 per cent total solids.[3] An equal volume of glacial acetic acid is added and thoroughly mixed by warming and shaking. The sirup is seeded, placed in an ice box overnight for crystallization to start, and is then frozen with an ice-salt mixture. The frozen mass is placed in a refrigerator at or near 0° C where it will thaw out slowly. After about a day the greater portion of the sugar will often have crystallized, but generally a week is required for complete crystallization. The yield is uniformly 42 to 45 per cent of the treated meal used.

WASHINGTON, December 12, 1921.

[3] Horton directs that the final sirup shall be concentrated to 96 per cent total solids. It is difficult and tedious to get such a thick mixture into solution in acetic acid, and it is obviously impractical where 2 or more kilos of mannose are to be made at one time. A concentration of 87 to 88 per cent total solids is amply heavy, and offers no difficulty either in dissolving in the acid or in subsequent crystallization.


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